# A Viscoplastic Model With Application to LiF-22%CaF<sub>2</sub> Hypereutectic Salt

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#### **Abstract**

A viscoplastic model for class M (metal-like behavior) materials is presented. One novel feature of this model is its use of internal variables to change the stress exponent of creep (where  $n \approx 5$ ) to that of 'natural' creep (where n = 3), in accordance with experimental observations. Another feature is the introduction of a coupling in the evolution equations of the kinematic and isotropic internal variables, making thermal recovery of the kinematic variable implicit. These features enable our viscoplastic model to reduce to that of steady-state creep in closed form. In addition, the hardening parameters associated with the two internal state variables (one scalar-valued, the other tensor-valued) are considered to be functions of state, instead of being taken as constant-valued. This feature enables each internal variable to represent a much wider spectrum of internal states for the material. We apply our model to a LiF-22%CaF2 hypereutectic salt, which is being considered as a thermal energy storage material for space-based solar dynamic power systems.

#### 1 Introduction

The primary power supply for Space Station Freedom will be photovoltaic panels (75 kW total-phase I implementation). Under consideration as an option, supplemental power will be supplied by reflecting the Sun's energy into two solar dynamic power modules (25 kW each—phase II implementation) [1]. Within the solar receiver of each power module will be a network of working fluid tubes. This working fluid will transport thermal energy to heat engines for electric power generation. Encasing each working fluid tube will be a string of canisters. The basic function of these canisters is to store heat when they are exposed to sunlight, and then release this heat when the vehicle is in eclipse. Typically, this energy storage-release cycle is attained through a thaw-freeze cycle of a special material contained within the canisters.

The attractive thermophysical properties of fluoride salts make them potential candidates for use as thermal energy storage materials in space-based solar dynamic systems [2]. Currently, the LiF-21mol%CaF<sub>2</sub> eutectic mixture is being considered for this purpose. A major problem that arises with the use of fluoride salts is the very large volume expansions that

they undergo upon melting ( $\sim 30\%$ ); plus, the fairly large volume expansions that they incur upon heating from room temperature to melting ( $\sim 5\%$ ). These volume expansions can lead to a stress buildup on the walls of a canister, especially when pockets of molten salt are entrapped between the containment vessel and unmelted salt. As a result, localized distortion or rupture of a canister could occur if the unmelted salt does not deform easily to accomodate this volume change.

This paper presents the development of a viscoplastic model for a LiF-22mol%CaF<sub>2</sub> hypereutectic salt based upon the experimental data of RAJ & WHITTENBERGER [3]. This viscoplastic model, and a similar one for the containment vessel (Haynes Alloy 188, a class A—alloy behavior, i.e. dynamic strain aging—material), will be used in future stress analyses of a canister to address the possiblity of localized distortion or rupture; thereby, assessing the durability of these canisters.

### 2 Stress-Strain Relations

Stress  $\sigma_{ij}$  is assumed to be related to infinitesimal strain  $\epsilon_{ij}$  through the isotropic constitutive equations

$$S_{ij} = 2\mu(e_{ij} - \varepsilon_{ij}^p) , \ \varepsilon_{ii}^p = 0$$
 (1)

$$\sigma_{ii} = 3\kappa(\epsilon_{ii} - \alpha \Delta T \delta_{ii}) \tag{2}$$

with shear  $\mu$  and bulk  $\kappa$  elastic moduli, where

$$S_{ij} = \sigma_{ij} - \sigma_{kk} \delta_{ij} / 3$$

$$e_{ij} = \epsilon_{ij} - \epsilon_{kk} \delta_{ij} / 3$$

denote the deviatoric stress and strain. Equation 1 characterizes the deviatoric stress response, while Eqn. 2 characterizes the hydrostatic stress response. The inelastic  $\varepsilon_{ij}^p$  and thermal  $\alpha \Delta T \delta_{ij}$  strains are eigenstrains that represent deviations from deviatoric and hydrostatic elastic behaviors, respectively. The

constant  $\alpha$  is the mean coefficient of thermal expansion, while  $\Delta T \equiv T - T_0$  represents a difference between the current temperature T and some reference temperature  $T_0$ . Herein, T represents an absolute temperature in degrees Kelvin. The quantity  $\delta_{ij}$  is the Kronecker delta. Repeated Latin indices are summed over from 1 to 3 in the usual manner.

Young's modulus E and Poisson's ratio  $\nu$  are often the elastic moduli that are experimentally determined. The expressions

$$\mu = \frac{E}{2(1+\nu)}$$

$$\kappa = \frac{E}{3(1-2\nu)}$$

define their interdependence, since only two elastic moduli are independent for isotropic materials.

## 3 Viscoplastic Theory

The mathematical structure for the theory of viscoplasticity considered in this paper is a special case of a much more general structure that was discussed by FREED & CHABOCHE [4]. The theory considered admits two internal state variables; they are: i) the (scalar-valued) yield strength Y > 0, and ii) the (deviatoric tensor-valued) back stress  $B_{ij}$ . The yield strength accounts for isotropic hardening effects, while the back stress accounts for kinematic (flow-induced anisotropic) hardening effects. These internal variables are considered to evolve phenomenologically through competitive processes associated with strain hardening, strain-induced dynamic recovery, and time-induced thermal recovery. The choice of a yield strength (over that of a drag strength) for the isotropic variable is consistent with results from an earlier study done by the authors [5].

The flow equation for inelastic strain is given by

$$\dot{\varepsilon}_{ij}^p = \frac{3}{2} ||\dot{\varepsilon}^p|| \frac{\Sigma_{ij}}{||\Sigma||} \tag{3}$$

with the effective stress

$$\Sigma_{ij} = S_{ij} - B_{ij}$$

establishing the direction of inelastic strain rate. A dot placed over a variable denotes its time rate-of-change.

The norms (or magnitudes) of this theory are defined as

$$||I|| = \sqrt{2/3 I_{ij} I_{ij}}$$
  
 $||J|| = \sqrt{3/2 J_{ij} J_{ij}}$ 

where  $I_{ij}$  is any deviatoric strain-like tensor, viz.  $\dot{\varepsilon}_{ij}^p$ , and where  $J_{ij}$  is any deviatoric stress-like tensor, viz.  $S_{ij}$ ,  $B_{ij}$  and  $\Sigma_{ij}$ . These norms are of the von Mises type, where the coefficients are chosen to scale the theory for tension.

The evolution equations characterizing the internal state of the material are given by

$$\dot{B}_{ij} = H\left(\frac{2}{3}\dot{\varepsilon}_{ij}^{p} - D\|\dot{\varepsilon}^{p}\|B_{ij} - R\frac{B_{ij}}{\|B\|}\right)$$
 (4)

and

$$\dot{Y} = h\left(\left\|\dot{\varepsilon}^p\right\| - d\left\|\dot{\varepsilon}^p\right\| Y - r\right) \tag{5}$$

where H>0 and h>0 are the strain hardening parameters,  $D\geq 0$  and  $d\geq 0$  are the dynamic recovery parameters, and  $R\geq 0$  and  $r\geq 0$  are the thermal recovery parameters. No parameter can be negative-valued and satisfy the thermodynamics, in general.

Defining functional forms for the six parameters H, h, D, d, R and r, along with a kinetic equation for  $||\varepsilon^p||$ , results in a specific viscoplastic model. Such a model is presented in the next section for class M materials.

## 4 Viscoplastic Model

Material functions that characterize a viscoplastic model for class M materials are given below. A discussion of these material functions is presented in the following subsection. Derivations of the material functions, where noted, are presented in the Appendix.

A Zener type kinetic equation for the evolution of inelastic strain is considered, i.e.

$$\|\dot{\varepsilon}^p\| = \vartheta Z \tag{6}$$

where  $\vartheta(T) > 0$  acts as a thermal diffusivity, and  $Z(S_{ij}, B_{ij}, Y) \ge 0$  is referred to as the Zener parameter. The Zener-Hollomon [6] hypothesis assumes that the kinetic equation can be represented as a product of two functions  $\vartheta$  and Z; the first is dependent only on temperature, while the latter is dependent only on stress and the internal state.

For the thermal diffusivity, we will use the relationship

$$\vartheta = \begin{cases} \exp\left(\frac{-Q}{kT}\right) \\ T_t \le T < T_m \\ \exp\left\{\frac{-Q}{kT_t} \left[\ln\left(\frac{T_t}{T}\right) + 1\right]\right\} \\ 0 < T \le T_t \end{cases}$$
 (7)

which was derived by MILLER [7]. Here k is the universal gas constant (k=8.314 J/mol-K), Q is the activation energy,  $T_m$  is the absolute melting temperature, and  $T_t$  is the absolute transition temperature. For the Zener parameter, we shall consider the relationship

$$Z = A \sinh^{3} \left( \frac{\langle ||\Sigma|| - Y \rangle}{K} \right)$$
 (8)

<sup>&</sup>lt;sup>1</sup>The preference of a temperature-dependent activation energy over a stress-dependent activation energy is discussed in a companion paper by FREED. RAJ & WALKER, which is also published in these conference proceedings.

with frequency coefficient A and drag strength K. The Macauley bracket operator  $\langle ||\Sigma|| - Y \rangle$  has either a value of 0 whenever  $||\Sigma|| \leq Y$  (defining the elastic domain), or a value of  $||\Sigma|| - Y$  whenever  $||\Sigma|| \geq Y$  (defining the viscoplastic domain), with  $||\Sigma|| = Y$  establishing the yield surface.

The three material parameters associated with the evolution of back stress for this particular model are taken to be

$$H = (0.01 + 0.99 \xi^{m}) E$$

$$D = \frac{y}{bY}$$

$$R = 0$$
(9)

where m and y are positive-valued material constants, and where b and  $\xi$  are functions of state defined in Eqns. 11 and 12. The function for dynamic recovery D is derived in the Appendix. Since R=0, there is no thermal recovery term in the evolution equation for back stress in this model.

The three material parameters associated with the evolution of yield strength for this particular model are taken to be

$$h = c_1 \sinh^{3-n} \left( \frac{Y}{yC} \right)$$

$$d = c_2 \frac{b\xi}{y}$$

$$r = A \vartheta \sinh^n \left( \frac{Y}{yC} \right)$$
(10)

where C,  $c_1$ ,  $c_2$  and n are positive-valued material constants. The function for thermal recovery r is derived in the Appendix. It is worth noting that unlike the recovery parameters, the functional form of a hardening parameter cannot be determined a priori, but must be postulated based upon experimental observations.

The fraction of applied stress which is attributed to the 'back stress' under steady-state

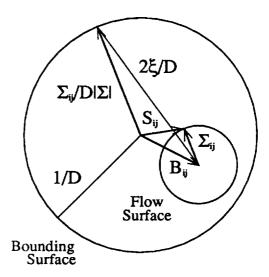


Figure 1: Geometric representation of state space.

conditions is determined to be

$$b = 1 - y - \frac{yK}{Y} \sinh^{-1} \left[ \sinh^{n/3} \left( \frac{Y}{yC} \right) \right]$$
 (11)

where the material constant y represents the percentage of applied stress which is attributed to the 'yield strength' under steady-state conditions. The derivation of Eqn. 11 is given in the Appendix.

A measure for the distance between the current point  $B_{ij}$  in stress space and its imaging point  $\Sigma_{ij}/D||\Sigma||$  on the bounding surface is given by

$$\xi = \sqrt{\frac{3}{8} \left( \frac{\Sigma_{ij}}{\|\Sigma\|} - D B_{ij} \right) \left( \frac{\Sigma_{ij}}{\|\Sigma\|} - D B_{ij} \right)}$$
(12)

where  $0 \le \xi \le 1$ . This relationship comes from considerations of state-space geometry for our model, as illustrated in Fig. 1. The cord between the current state of back stress and its

image point of length  $2\xi/D$  is in the direction that the back stress evolves, which is the viscoplastic analog to the kinematic evolution in a two-surface MROZ [8] model for plasticity. A value of  $\xi = 0$  implies that  $B_{ij}$  is on the bounding surface, and that  $S_{ij}$  is either on this surface or outside of it. A value of  $\xi = 1$  also implies that  $B_{ij}$  is on the bounding surface, but now  $S_{ij}$  is inside this surface, i.e. unloading from a saturation state has just begun.

#### 4.1 Discussion of the Model

In total, there are three temperaturedependent material constants pertaining to thermoelasticity, viz.  $\alpha$ ,  $\kappa$  and  $\mu$ , ten temperature-independent material constants pertaining to viscoplasticity, viz.  $A, C, c_1, c_2$ ,  $K, m, n, Q, T_t$  and y, and one initial condition, viz. Yo. Of these ten viscoplastic constants, five are determined from steady-state creep data, viz. A, C, n, Q and  $T_t$ , while the remaining five must be determined from data involving transient behavior. By definition,  $Y_0 = 0$  in an annealled state. The material constants for LiF-22%CaF2 used in the analyses presented in this paper are listed in Table 1. Data for the elastic constants [9] presented therein were correlated over the temperature range of 300 to 950 K, with melting occurring at 1042 K. There is danger in extrapolating their values outside of this range, especially for  $\nu$  whose expression is in direct violation with physics for T < 150 K.

An important consequence of the viscoplastic model presented above is that the Zener parameter defined in Eqn. 8 reduces in closed form to the Zener parameter associated with Garofalo's [10] creep equation under steady state conditions, *i.e.* 

$$Z_{ss} = A \sinh^n \left( \frac{\|S\|}{C} \right) \tag{13}$$

where the subscript 'ss' denotes steady state,

Constant	Units	Value
	10 <sup>6</sup> K <sup>-1</sup>	L
α		18 + 0.03T
μ	MPa	52,000 - 29T
ν	_	0.65 - 0.001T
A	$s^{-1}$	$5 \times 10^{15}$
C	MPa	27
$c_1$	MPa	50,000
$c_2$	_	0
K	MPa	8
$m_{\cdot}$		1
n		5
Q	J/mol.	320,000
$T_m$	K	1042
$T_{t}$	K	850
y	_	0.1
$Y_0$	MPa	0

Table 1: Constants for LiF-22%CaF<sub>2</sub>.

and C is the power-law breakdown stress. The capability of Zener's kinetic equation (6) to correlate experimental creep data using the relationships for  $\vartheta$  and  $Z_{ss}$  given in Eqns. 7 and 13 is demonstrated in Fig. 2.

The hardening parameter H given in Eqn. 9 is similar in form and function to the tangent modulus of the Dafalias-Popov [11] two-surface theory of plasticity. Both allow for a smooth transition between elastic and inelastic behavior. The exponent m characterizes the strength of curvature in a typical hysteresis loop. Since there are no cyclic data available for LiF-22%CaF<sub>2</sub>, an assumed value for m is given in Table 1.

As previously mentioned, there is no 'explicit' representation for the thermal recovery of back stress in our model. But take note that the surface which bounds the state of back stress—characterized by D—is a function of the yield strength in Eqn. 9. Consequently, since the yield strength thermally re-

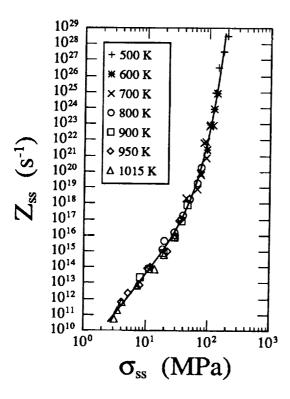


Figure 2: Steady-state kinetic response of LiF-22%CaF<sub>2</sub>. Data are from RAJ & WHIT-TENBERGER [3].

covers, thermal recovery of the back stress is 'implicit'. Unfortunately, inelastic strain must evolve in order for this implicit recovery to take place. Even though this is not a physically accurate representation of thermal recovery, it is a very useful approximation to it. It is useful because it allows the effect of thermal recovery to be modeled, while still enabling our viscoplastic model to reduce to a steady-state creep model (in particular, Garofalo's) in closed form. This enables us to use creep data directly for the purpose of characterizing about half of our model's material constants—a huge benefit.

In the domain of power-law creep, the hardening and thermal recovery parameters, h and r, given in Eqn. 10 are similar in form and function to the hardening/recovery relationships proposed and experimentally verified by MITRA & MCLEAN [12]. Here the stress dependence of recovery, as defined by the product hr, has an exponent of 3. This is in accordance with the 'natural' theory of creep for thermally-assisted dislocation climb [13]. Our particular hardening and thermal recovery parameters extrapolate the Mitra-McLean results into the domain of exponential behavior. The viscoplastic model of Robinson [14] also employs the hardening/recovery concept of Mitra and McLean, but in the evolution equation for back stress.

In the viscoplastic model of MILLER [7], the dynamic recovery parameter d was introduced to provide a coupling between the back stress and drag strength so that differences between the monotonic and cyclic stress-strain curves could be accounted for. This distinction between monotonic and cyclic behavior is a phenomenon that Chaboche et al. [15] refer to as the strain memory effect, but one that they model in a completely different manner than the approach used by Miller. Our particular function for d given in Eqn. 10 differs from Miller's in form, but not in purpose. In both of

these models,  $d_{ss}=0$  at steady state, thereby enabling the viscoplastic models to reduce to well-known creep models in closed form. In our model,  $\xi$  (not ||B||) is the parameter chosen to account for the oscillatory changes in of the internal state of the material during cyclic loadings. The ratio b/y proportions this coupling according to the relative strength capability of the back stress to yield strength. Because there are no cyclic data available for LiF-22%CaF<sub>2</sub>, this coupling effect is not considered in this paper.

The material constant y establishes, among other things, the capability of a material to cyclically harden. The greater the value of y, the greater is this capability. An assumed value for y is given in Table 1 for LiF-22%CaF<sub>2</sub> because of a lack of cyclic data.

The expression for b in Eqn. 11 (defining that portion of the applied stress which is attributed to the back stress under conditions of steady state) is a direct consequence of the following hypothesis: it is the internal state of stress that accounts for the difference between 'natural' (with a stress exponent of 3, cf. Eqn. 8) and observed (where  $n \approx 5$ , cf. Eqn. 13) power-law creep behavior in class M materials. This internal stress results from a honeycomb-like dislocation structure composed of hard cell walls of high dislocation density which surround soft cell interiors of relatively low dislocation density [16]. The 'natural' theory of creep assumes a homogeneous dislocation network. Notice that when n = 3(as is the case for class A materials, which typically have no cellular dislocation structure), the fraction of back stress to applied stress at steady state becomes constant-valued; in particular,

$$b = 1 - y - \frac{K}{C}$$

Otherwise, b varies with material strength, for which the yield strength Y is an appropriate measure. It is this feature of the variable b that

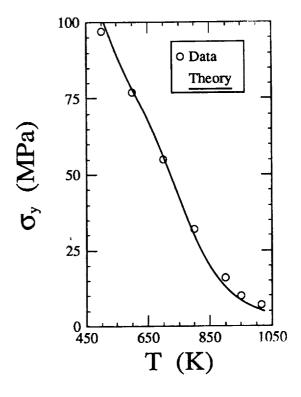


Figure 3: Yield strength (0.2% offset) of LiF-22%CaF<sub>2</sub>.  $\dot{\epsilon} = 2 \times 10^{-4} \ s^{-1}$ . Data are from RAJ & WHITTENBERGER [3].

distinguishes this viscoplastic model from the previous ones developed by the authors [5, 17].

## 5 Applications

There are very few experimental data for  $\text{LiF-}22\%\text{CaF}_2$  that one can use to validate a model such as the one given above. About the only data that are available, besides those which appear in Fig. 2, are yield strength data. Figure 3 presents the predictive capability of our model, as it pertains to the yield strength. The yield strength  $\sigma_y$  at 500 K, and the fact that about 15% strain was required to attain

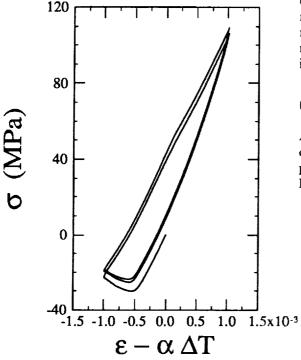


Figure 4: Out-of-phase TMF response predicted for LiF-22%CaF<sub>2</sub>.  $T_{\rm min} = 400$  K.  $T_{\rm max} = 800$  K.  $\dot{\epsilon}_{\rm mech} \equiv \dot{\epsilon} - \alpha \, \dot{T} = 1 \times 10^{-4} \, s^{-1}$ .

steady-state in this test, were facts used to set values for the material constants K and  $c_1$ , respectively. The model predictions for all other temperatures are just that, predictions. The agreement between theory and experiment is quite good.

To demonstrate some of the capabilities of our model, the response to the first few cycles of a typical thermomechanical fatigue (TMF) loading history are presented in Fig. 4. Unfortunately, there are no experimental data to substantiate this prediction one way or the other. Nevertheless, the predicted shape of this hysteresis loop is consistent with shapes

observed from similar experiments done on metals [18]. In short, our model is capable of representing the complexities of nonisothermal material behavior while using temperatureindependent viscoplastic material constants.

## 6 Conclusions

A viscoplastic model has been proposed for class M materials. This model has been applied to a LiF-22%CaF<sub>2</sub> hypereutectic salt. Features of this model include:

- Closed form reduction to creep theory under steady-state conditions.
- Internal state variables account for the difference in the stress dependence of 'natural' and observed creep behavior.
- Two internal state variables are employed.
  - The yield strength accounts for isotropic hardening and softening effects.
  - 2. The back stress accounts for kinematic hardening and softening effects.
- These internal variables evolve according to the competitive processes of strain hardening, and dynamic and thermal recovery.
- A Mitra-McLean hardening/recovery format is used in the evolution of yield strength.
- Dynamic recovery of the yield strength accounts for monotonic/cyclic interaction (or strain-memory) effects.
- Thermal recovery of the back stress is taken to be implicit; thereby, simplifying the structure and characterization procedure of the model.

 The strain hardening parameter for back stress is comparable with the tangent modulus in the Dafalias-Popov twosurface theory of plasticity.

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## Appendix

Much of the development of our viscoplastic model is predicated upon the assumption that

$$||B||_{ss} = b ||S|| \text{ and } Y_{ss} = y ||S||$$
 (14)

where y is taken to be a constant, but not b. It also makes use of the fact that the back stress  $B_{ij}$  is coaxial with the deviatoric stress  $S_{ij}$  under steady-state conditions (a direct consequence of Eqns. 3 and 4, and in agreement with experimental evidence [19]), and therefore one can write

$$||\Sigma||_{ss} \equiv ||S - B||_{ss} = ||S|| - ||B||_{ss}$$
 (15)

which is not valid, in general.

By definition,  $\dot{B}_{ij} = 0$  at steady state, and since R = 0 Eqn. 4 reduces to

$$D_{ss} = \frac{1}{||B||_{ss}}$$

Combining this result with Eqn. 14 leads directly to the dynamic recovery parameter given in Eqn. 9. Because D is a strength measure (it establishes the magnitude of the limiting state of back stress), it is appropriate that it depend on the yield strength and not on the magnitude of back stress.

Likewise,  $\dot{Y} = 0$  at steady-state by definition, and therefore

$$r_{ss} = ||\dot{\varepsilon}^p||_{ss} = \vartheta Z_{ss}$$

where we make use of the self-imposed constraint that  $d_{ss} = 0$  (satisfied by the fact that  $\xi_{ss} = 0$ ). Combining Eqns. 13 and 14 with this result leads to the thermal recovery parameter given in Eqn. 10.

Equating the transient Zener parameter of Eqn. 8 with its steady-state counterpart given in Eqn. 13 under steady-state conditions, and using Eqns. 14 and 15, one obtains the relationship

$$b + y = 1 - \frac{K}{\|S\|} \sinh^{-1} \left[ \sinh^{n/3} \left( \frac{\|S\|}{C} \right) \right]$$
(16)

which when written in terms of  $Y_{ss}$  via Eqn. 14 results in Eqn. 11 for b. Here  $\sinh^{-1}$  is the arcsinh, whose equivalent expression

$$\sinh^{-1}(x) \equiv \ln\left(x + \sqrt{x^2 + 1}\right)$$

is a useful representation when writing For-TRAN code.

A graphic representation of Eqn. 16 is presented in Fig. 5. In the domain of power-law creep, Eqn. 16 reduces to a relationship that ČADEK [20] proposed and experimentally verified, where b and y were not distinguished one from the other. In the domain of exponential creep, Eqn. 16 implies that the fraction of internal stress (b+y)||S|| to applied stress ||S||at steady state is nearly a constant. The ratio K/C establishes this plateau, which suggests an upper bound for y. As a rule of thumb, a ratio of  $K/C \approx 1/3$  leads to an internal stress that is between 40 and 50% of the applied stress at steady state, which is consistent with experimental observations [21]. In this study, the value of K was determined by correlating yield strength data, and was found to agree with this rule of thumb.

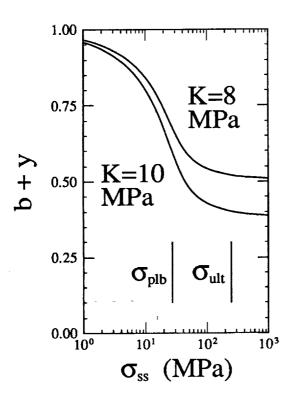


Figure 5: Response of Eqn. 16, where values for the power-law breakdown stress  $\sigma_{\text{plb}} \equiv C$ , the ultimate strength  $\sigma_{\text{ult}}$  and the creep exponent n pertain to LiF-22%CaF<sub>2</sub>. Plots for two different values of K are displayed.

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	A viscoplastic model for class M (met its use of internal variables to change $n=3$ ), in accordance with experiment evolution equations of the kinematic at variable implicit. These features enable form. In addition, the hardening paramother tensor-valued) are considered to feature enables each internal variable tapply our model to a LiF-22%CaF <sub>2</sub> h material for space-based solar dynamic	the stress exponent aql observations. And isotropic internal e our viscoplastic meters associated with be functions of state to represent a much sypereutectic salt, where the stress is the stress of	of creep (where n a nother feature is the variables, making odel to reduce to the the two internal se, instead of being wider spectrum of	≈ 5) to that of 'nature introduction of a continuous control of a continuous control of a control of steady-state control of state variables (one taken as constant-vainternal states for the	ral' creep (where coupling in the the kinematic reep in closed scalar-valued, the lued. This he material. We			
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